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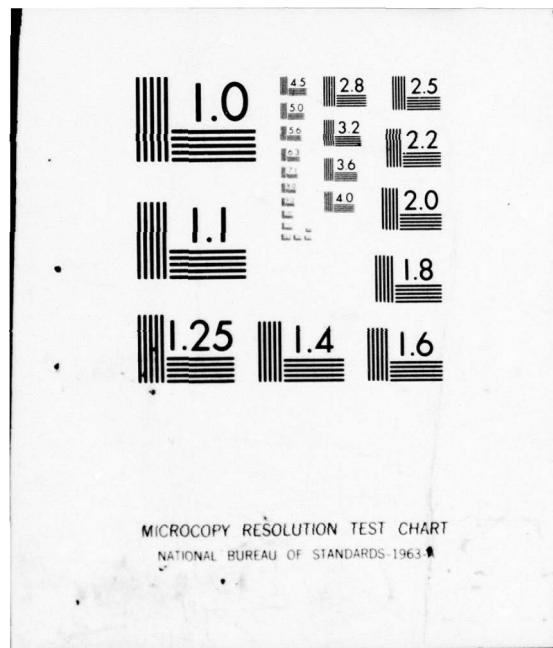
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6 Reactions of Metal-to-Metal Multiple Bonds. 5.

9 Addition of Nitric Oxide to Hexakis(tert-butoxy)-ditungsten.

Preparation, Properties and Structural Characterization of

10 Tris(tert-butoxy)(nitrosyl)(pyridine)tungsten.

M. H. Chisholm¹, F. A. Cotton², M. W. Extine²

R. L. Kelly²

Departments of Chemistry

¹Princeton University

Princeton, New Jersey 08540

and

²Texas A & M University

College Station, Texas 77843

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The reaction between $W_2(OBu^t)_6$ and nitric oxide (2 equiv) in hydrocarbon solvents yields an insoluble pale-yellow product of empirical formula $W(OBu^t)_3NO$ ($\nu_{NO} = 1560 \text{ cm}^{-1}$). Addition of the nitrogen donor ligands NH_3 and pyridine causes the above compound to dissolve with the formation of mononuclear compounds $W(OBu^t)_3(NO)(L)$. The yellow crystalline compound $W(OBu^t)_3(NO)(C_5H_5N)$ ($\nu_{NO} = 1555 \text{ cm}^{-1}$) has been obtained directly by the reaction between $W_2(OBu^t)_6$ and NO (2 equiv) in pyridine as the solvent. The compound crystallizes in the		

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space group $P2_1/n$ with $Z=4$ and unit cell dimensions $a = 9.694(2)$, $b=15.686(3)$, $c=14.358(2)\text{\AA}$, $\beta=97.40(1)^\circ$ and $V=2165.1(7)\text{\AA}^3$. The coordination geometry of the WO_3N_2 moiety is a slightly distorted trigonal bipyramidal with the axial positions occupied by the nitrogen atoms of the nitrosyl and pyridine ligands. The tungsten atom is displaced 0.34\AA towards the nitrosyl ligand from the equatorial plane of the three alkoxy oxygen atoms. There is a linear W-N-O moiety with a short W-N bond distance, $1.732(8)\text{\AA}$, whereas the W-N bond distance to the coordinated pyridine is long, $2.323(7)\text{\AA}$.

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Reactions of Metal-to-Metal Multiple Bonds. 5.¹
Addition of Nitric Oxide to Hexakis(tert-butoxy)-ditungsten.
Preparation, Properties and Structural Characterization of
Tris(tert-butoxy)(nitrosyl)(pyridine)tungsten.

M. H. Chisholm*^{2a}, F. A. Cotton*^{2b}, M. W. Extine^{2b}
and R. L. Kelly^{2a}

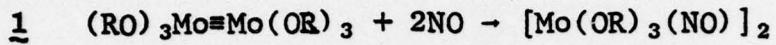
Contribution from the Departments of Chemistry, Princeton University, Princeton, New Jersey 08540 and Texas A and M University, College Station, Texas 77843.

Abstract

The reaction between $W_2(OBu^t)_6$ and nitric oxide (2 equiv) in hydrocarbon solvents yields an insoluble pale-yellow product of empirical formula $W(OBu^t)_3NO$ ($\nu_{NO} = 1560 \text{ cm}^{-1}$). Addition of the nitrogen donor ligands NH_3 , NMe_3 and pyridine causes the above compound to dissolve with the formation of mononuclear compounds $W(OBu^t)_3(NO)(L)$. The yellow crystalline compound $W(OBu^t)_3(NO)(C_5H_5N)$ ($\nu_{NO} = 1555 \text{ cm}^{-1}$) has been obtained directly by the reaction between $W_2(OBu^t)_6$ and NO (2 equiv) in pyridine as the solvent. The compound crystallizes in the space group $P2_1/n$ with $Z=4$ and unit cell dimensions $a = 9.694(2)$, $b = 15.686(3)$, $c = 14.358(2)\text{\AA}$, $\beta = 97.40(1)^\circ$ and $V = 2165.1(7)\text{\AA}^3$. The coordination geometry of the WO_3N_2 moiety is a slightly distorted trigonal bipyramidal with the axial positions occupied by the nitrogen atoms of the nitrosyl and pyridine ligands. The tungsten atom is displaced 0.34\AA towards the nitrosyl ligand from the equatorial plane of the three alkoxy oxygen atoms. There is a linear $W-N-O$ moiety with a short $W-N$ bond distance, $1.732(8)\text{\AA}$, whereas the $W-N$ bond distance to the coordinated pyridine is long, $2.323(7)\text{\AA}$.

Introduction

Previously we have shown that the molybdenum-to-molybdenum triple bond in the dinuclear alkoxides $\text{Mo}_2(\text{OR})_6$ ³ is cleaved in reaction 1.⁴



where R=Me₃C, Me₂CH and Me₃CCH₂

The structural characterization of $[\text{Mo}(\text{OPr})_3\text{NO}]_2$ ¹ revealed two equivalent (inversion-related) distorted trigonal bipyramidal $\text{Mo}(\text{OR})_4\text{NO}$ units fused along a common axial-to-equatorial edge through the agency of bridging iso-propoxy groups. With a Mo-to-Mo distance of 3.335(2) Å it can be safely assumed that no direct metal-to-metal bonding exists.⁵ In a formal sense reaction 1 corresponds to the replacement of the metal-to-metal triple bond by two metal-to-ligand triple bonds followed by Lewis base association.⁶

We concluded⁴ that "There would not seem to be any reason why discrete mononuclear complexes of type A, where X represents a univalent ligand, L a sigma donor, and M any atom or ion isoelectronic with Mo(III), should not exist as a general class."



We report here our preparation and characterization of the first member of this class, namely $\text{W}(\text{OBu}^t)_3(\text{NO})(\text{C}_5\text{H}_5\text{N})$, formed in the reaction between $\text{W}_2(\text{OBu}^t)_6$ and NO (2 equiv) in pyridine.

Results and Discussion

Synthesis. Addition of nitric oxide (2 equiv) to hydrocarbon solutions of $\text{W}_2(\text{OBu}^t)_6$ ⁷ leads to the formation of a fine yellow

precipitate of empirical formula $W(OBu^t)_3NO$ which shows a single sharp and very strong i.r. absorption at 1565cm^{-1} assignable to ν_{NO} . This compound is virtually insoluble in alkane and aromatic hydrocarbons which has hindered its further characterization. It is believed to be polymeric, $[W(OBu^t)_3NO]_n$, in contrast to the dimeric molybdenum analogue $[Mo(OBu^t)_3NO]_2$. $[W(OBu^t)_3NO]_n$ will dissolve in the presence of Me_3N and pyridine yielding $W(OBu^t)_3 - (NO)(L)$. The compound $W(OBu^t)_3(NO)(C_5H_5N)$ has also been made directly by the addition of NO (2 equiv) to a pyridine solution of $W_2(OBu^t)_6$ and is a yellow crystalline compound appreciably soluble in hydrocarbon solvents. For $W(OBu^t)_3(NO)(C_5H_5N)$ a strong sharp i.r. absorption at 1555cm^{-1} is assignable to ν_{NO} ; a sharp band of medium intensity at 1610cm^{-1} is assigned to the stretching vibration of the coordinated pyridine. The nmr spectra in toluene- d_8 of $W(OBu^t)_3(NO)(C_5H_5N)$ show the presence of only one type of tert-butoxy ligand, even at -60°C . This, together with the observation of a single nitrosyl stretching frequency, is consistent with the presence in solution of a structure akin to that found in the crystal.

Solid State Structure of $W(OBu^t)_3(NO)(C_5H_5N)$. The compound is composed of discrete mononuclear molecules in the solid state. Atomic positional and thermal parameters are given in Table I. The molecular structure is shown in Figure 1 along with the atom labelling scheme. Bond distances and angles are given in Table II. As can be seen from Figure 1, the coordination geometry is a slightly distorted trigonal bipyramidal with the axial positions occupied by the nitrosyl and pyridine ligands.

The tungsten atom is displaced 0.34\AA towards the nitrosyl ligand from the equatorial plane of the three alkoxy oxygen atoms.

The nitrosyl ligand is coordinated linearly and the W-N1 bond is quite short, $1.732(8)\text{\AA}$, indicative of some multiple bond character whereas the tungsten to pyridine bond is considerably longer, $W\text{-N2}=2.323(7)\text{\AA}$. The W-O distances are in the expected range.

Bonding. The trigonal set of ligands splits the tungsten 5d orbitals into three sets: a (d_{z^2}), e ($d_{x^2-y^2}$, d_{xy}) and e (d_{xz} , d_{yz}). The second e set is but little involved in metal ligand σ -bonding and thus lies lowest in energy. In $W(\text{OBu}^t)_3(\text{NO})(\text{py})$ tungsten achieves only a fourteen valence shell electronic configuration. Ten electrons are involved in forming the five σ bonds and the remaining four occupy the lower e orbitals which have the appropriate symmetry to interact with the empty nitrosyl π^* orbitals. The bonding is analogous to that in $[\text{Mo}(\text{OPr}^1)_3\text{NO}]_2$, where a bridging isopropoxide occupies the axial position trans to the nitrosyl ligand.

For a linear metal nitrosyl moiety, metal-to-nitrosyl π^* bonding should be reflected in (i) the metal-to-nitrogen bond distance, (ii) the nitrogen-to-oxygen bond distance and (iii) the value of the N-O stretching frequency. A lengthening of the N-O bond and a lowering of $\nu(\text{NO})$ should correlate with an increase in metal-to-nitrosyl π^* -bonding. The shortness of the metal-to-nitrogen bond may also correlate with M-N multiple bond character but this distance will also be influenced by the nature of the metal σ -hybrid orbital used in forming the M-N bond. The latter is determined by the coordination number and geometry of the metal complex as well as by the nature of the other ligands bonded to the metal.

M-N and N-O bond distances and $\nu(\text{NO})$ values for some compounds containing linear M-N-O moieties are given in Table 3 and are

illustrative of the above considerations. Certainly little can be inferred from the M-N distances alone. There does, however, seem to be the expected correlation between N-O bond length and $\nu(\text{NO})$. This correlation is limited, however, by the relatively small changes and large experimental errors which occur in N-O distances. One can conclude that the generally accepted view that $\nu(\text{NO})$ correlates with metal-to-nitrosyl π^* bonding finds structural support in N-O bond distances.

The trihaloruthenium complexes, which contain six coordinate metal atoms, all show very similar M-N-O parameters (see Table 3). Notably the values of $\nu(\text{NO})$ are more than 200 cm^{-1} higher, and the N-O distances are significantly shorter than those of the five coordinate molybdenum and tungsten complexes. The metals here are all in the +2 oxidation state, if we assume the formalism $\text{M}^-(\text{NO})^+$ for the linear M-N-O moiety. Evidently the $(t_{2g})^6$ -to- $\text{NO}\pi^*$ bonding is less effective in these Ru(2+) octahedral complexes than is the $(e)^4$ -to- $\text{NO}\pi^*$ bonding in the Mo(2+) and W(2+) trigonal bipyramidal complexes, despite the presence of only 14-valence shell electrons in the latter. A plausible rationale for this observation may lie in the mixing of ligand-to-metal π -bonding (p-d) and metal-to- $\text{NO}\pi^*$ bonding. Ligand (OR or NR_2)-to-metal- π bonding in the four-coordinate $\text{Cr}(\text{NR}_2)_3\text{NO}$ and five-coordinate $\text{M}(\text{OR})_3(\text{NO})\text{L}$ molecules will raise the energy of the filled metal d_{xz} and d_{yz} atomic orbitals from the level they would otherwise have had as a result of pure M-L σ -bonding. The energy separation between filled metal d_{xz} and d_{yz} orbitals and the vacant higher energy $\text{NO}\pi^*$ orbitals will be reduced and metal-to-nitrosyl π^* bonding enhanced. It is, of course, not possible to separate completely the σ and π -donor properties of a ligand. However, the values of $\nu(\text{NO})$ do go down as the overall donor ($\sigma + \pi$) properties of the ligand increase: compare $\nu(\text{NO})=1698 \text{ cm}^{-1}$ for

$\text{Cr}(\text{N}(\text{SiMe}_3)_2)_3\text{NO}$ with $\nu(\text{NO})=1640 \text{ cm}^{-1}$ for $\text{Cr}(\text{NPr}^1_2)_3\text{NO}$ and $\nu(\text{NO})=1643$, 1640 and 1630 cm^{-1} for the compounds $[\text{Mo}(\text{OR})_3\text{NO}]_2$ where $\text{R} = \text{CH}_2\text{CMe}_3$, CHMe_2 and CMe_3 , respectively.

Experimental Section

General procedures have been described;¹ note the use of dry and oxygen free atmospheres and solvents.

$\text{W}_2(\text{OBu}^t)_6$ was prepared from the reaction between $\text{W}_2(\text{NMe}_2)_6$ and Bu^tOH (> 6 equiv) in benzene and was recrystallized from hexane solutions.⁷

$[\text{W}(\text{OBu}^t)_3\text{NO}]_n$: $\text{W}_2(\text{OBu}^t)_6$ (0.63g, 0.78 mmol) was dissolved in hexane (10 mL) to give a red solution. Nitric oxide (1.56 mmol) was added with the use of a calibrated manifold to the above solution frozen at liquid nitrogen temperature which yielded upon warming to room temperature a green solution and a pale yellow precipitate. After 5h the pale yellow precipitate was collected by filtration, washed with hexane, and dried in vacuo (10^{-4} cm Hg , 25°C). Yield 0.56g (83% based on tungsten). Analysis found (calcd) for $\text{W}(\text{OBu}^t)_3\text{NO}$: C, 33.06 (33.27); H, 6.20 (6.28); N, 3.40 (3.23).

I.r. data obtained from a nujol mull between CsI plates (2000-300 cm^{-1} range): 1565 vs, 1310 w, 1245 m, 1165 s (broad), 1090 s, 1030 w, 948 vs (broad), 928 vs, 912 s, 796 m (sharp), 784 m (sharp), 724 m (broad), 627 s (sharp), 595 m, 572 m, 485 w, 394 w, 381 w, 340 w.

$\text{W}(\text{OBu}^t)_3(\text{NO})(\text{C}_5\text{H}_5\text{N})$: $\text{W}_2(\text{OBu}^t)_6$ (0.1844g, 0.23 mmol) was dissolved in pyridine (7 ml) to form a deep red solution. This was frozen at liquid nitrogen temperature and nitric oxide (0.46 mmol) was added using a calibrated vacuum manifold. The reaction mixture was allowed to warm to room temperature and left to stand for 12h. The pyridine was stripped and the residue extracted with

toluene (ca. 5 mL). The pale yellow solution was filtered to remove a small amount of a black insoluble material. The filtrate was collected and cooled to -10°C yielding pale yellow crystals (ca. 80 mg). Analysis found (calcd) for $W(OBu^t)_3(NO)(C_5H_5N)$: C, 39.65 (39.86); H, 6.25 (6.30); N, 5.40 (5.47). I.r. data obtained from a nujol mull using CsI plate in the range 2000-300 cm^{-1} : 1610 m (sharp), 1555 vs, 1305 w, 1240 m, 1222 m (sharp), 1170 m (broad), 1156 m (sharp), 1076 m (sharp), 1043 m (sharp), 1027 w, 1018 w, 1000 w, 965 m, 948 m, 937 vs (broad), 910 m, 900 w, 783 m, 762 m (sharp), 722 m (broad), 694 m (sharp), 621 s, 576 m, 485 w, 436 w, 380 w.

^{13}C nmr data obtained in toluene- d_8 at -50°C: $\delta(\text{OC}) = 80.8$, $\delta(\text{CH}_3) = 32.9$. (δ in ppm rel. TMS).

X-Ray Crystallography.⁸ A yellow crystal of $W(OBu^t)_3(NO)(C_5H_5N)$ measuring 0.23x0.28x0.58 mm was mounted, embedded in epoxy cement and sealed in a thin walled glass capillary, with its longest dimension nearly coincident with the phi axis. Omega scans of several intense low-angle reflections had peak widths at half height of ca. 0.2°. Cell constants and axial photographs indicated that the crystal belonged to the monoclinic system with $a = 9.694(2)$, $b = 15.686(3)$, $c = 14.358(2)\text{\AA}$, $\beta = 97.40(1)^\circ$, and $V = 2165.1(7)\text{\AA}^3$. The observed volume was consistent with that expected for $Z = 4$. Systematic absences observed during data collection, on oko ($k = 2n+1$) and hol ($h+l = 2n+1$), uniquely determined the space group to be $P2_1/n$ (a non-standard setting of $P2_1/c$, No. 14).

The data were collected at $23 \pm 2^\circ\text{C}$ with a Syntex PI autodiffractometer equipped with a graphite crystal monochromator and using $\text{MoK}\alpha$ ($\lambda = 0.710730\text{\AA}$) radiation. Variable scan rates from 4.8 to 24.0°/min were used for symmetric $\theta/2\theta$ scans ranging from 1.0° below to 1.0° above the calculated $\text{MoK}\alpha_1, \text{K}\alpha_2$ doublet. The

ratio of background to scan time was 0.5. A total of 2936 unique reflections having $0^\circ < 2\theta \text{MoK}\alpha < 45^\circ$ were collected. The intensities of three standard reflections were monitored frequently throughout data collection and showed an approximately linear decrease of 12% over the period of data collection. The data were reduced to a set of relative $|F_o|^2$ values and then corrected for crystal decay. An empirical absorption correction based upon a series of psi scans was applied to the data ($\mu = 56.7 \text{ cm}^{-1}$); relative transmission factors ranged from 0.844 to 1.000 with an average of 0.949. The 2103 observations having $|F| > 3\sigma(|F|)$ were retained as observed and used in subsequent structure solution and refinement.

The positions of the 24 unique non-hydrogen atoms were determined by standard heavy atom methods. The structure was refined to convergence using anisotropic thermal parameters for all 24 atoms. The final discrepancy indices were

$$R_1 = \sum | |F_o| - |F_c| | / |F_o| = 0.032$$

$$R_2 = [\sum w (|F_o| - |F_c|)^2 / \sum w |F_o|^2]^{1/2} = 0.047$$

The estimated standard deviation of an observation of unit weight was 1.102. The top peaks in a final difference Fourier map were due to methyl group hydrogen atoms.

A table of observed and calculated structure factors (9 pages) is available as supplementary material. See any current masthead page for ordering information.

Acknowledgements

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References

1. Part 4. W. I. Bailey, M. H. Chisholm, F. A. Cotton and L. A. Rankel, J. Am. Chem. Soc., submitted for publication.
2. a) Princeton University b) Texas A&M University.
3. M. H. Chisholm, F. A. Cotton, C. A. Murillo and W. W. Reichert, Inorg. Chem., 16, 1801 (1977).
4. M. H. Chisholm, F. A. Cotton, M. W. Extine and R. L. Kelly, J. Am. Chem. Soc. 100 xxx (1978).
5. A good comparison can be made between two structurally related compounds $\text{Mo}_2(\text{OPr}^i)_6(\text{NO})_2$ and $\text{Mo}_2(\text{OPr}^i)_8$. The latter has a Mo-to-Mo distance of $2.525(2)\text{\AA}$ indicative of a metal-to-metal double bond. M. H. Chisholm, F. A. Cotton, M. W. Extine and W. W. Reichert, Inorg. Chem. submitted for publication.
6. M. H. Chisholm, Advances in Chemistry Series, in press.
7. M. H. Chisholm and M. W. Extine, J. Am. Chem. Soc. 97, 5626 (1975).
8. All crystallographic computations were carried out using the Enraf Nonius Structure Determination Package and a PDP11/45 computer owned by Molecular Structure Corp., College Station, Texas.

TABLE I. POSITIONAL AND THERMAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS.

ATOM	X	Y	Z	$\theta(1.1)$	$\theta(2.2)$	$\theta(3.3)$	$\theta(1.2)$	$\theta(1.3)$	$\theta(2.3)$
W	0.05941(4)	0.23082(3)	0.08295(2)	2.90(1)	3.59(2)	2.80(1)	-0.09(1)	0.56(1)	0.45(1)
O1	-0.0626(6)	0.1482(4)	0.0250(4)	3.9(3)	4.4(3)	3.3(3)	-0.4(3)	-0.5(2)	0.4(3)
O2	0.2467(6)	0.1974(4)	0.1196(4)	3.2(3)	4.8(3)	3.5(3)	-0.0(2)	0.9(2)	0.7(2)
O3	-0.0194(6)	0.3097(4)	0.1602(4)	4.1(3)	3.6(3)	3.8(3)	0.0(3)	1.3(2)	0.7(2)
O4	0.0918(8)	0.3428(5)	-0.0806(5)	7.0(4)	5.7(4)	4.5(3)	0.6(3)	2.1(3)	2.6(3)
N1	0.0779(8)	0.2964(5)	-0.0117(5)	4.0(3)	4.5(4)	4.1(4)	0.4(3)	1.0(3)	1.1(3)
N2	0.0280(7)	0.1486(5)	0.2131(5)	2.8(3)	4.1(3)	3.1(3)	-0.3(3)	0.7(3)	0.1(3)
C1	-0.1302(11)	0.1312(7)	-0.0713(7)	5.4(5)	4.9(5)	3.0(4)	-0.1(4)	-0.8(4)	-0.6(4)
C2	-0.0146(13)	0.1150(9)	-0.1326(8)	8.0(7)	8.8(7)	3.3(5)	1.5(6)	-0.3(5)	-1.7(5)
C3	-0.2176(13)	0.2086(7)	-0.1062(9)	6.3(6)	4.9(6)	6.7(6)	0.8(5)	-0.7(5)	1.2(5)
C4	-0.2203(13)	0.0515(8)	-0.0594(9)	7.1(7)	6.0(6)	6.5(6)	-2.6(5)	-2.1(6)	0.2(5)
C5	0.3771(10)	0.2141(7)	0.0837(8)	3.2(4)	6.5(7)	5.4(5)	-0.3(4)	1.4(4)	0.3(5)
C6	0.4030(12)	0.3098(9)	0.0875(9)	6.2(6)	6.8(6)	8.5(7)	-2.7(5)	2.5(5)	-1.0(6)
C7	0.3682(11)	0.1819(9)	-0.0177(7)	5.2(5)	9.0(7)	4.1(5)	-0.2(6)	1.9(4)	-1.5(5)
C8	0.4869(11)	0.1626(10)	0.1500(9)	3.1(5)	12(1)	6.0(6)	1.0(6)	0.1(5)	1.0(7)
C9	-0.0600(10)	0.3984(6)	0.1535(7)	4.2(4)	3.4(4)	4.4(4)	-0.0(4)	1.2(4)	0.6(4)
C10	0.0691(11)	0.4526(7)	0.1492(8)	4.9(5)	4.2(5)	6.6(6)	-0.6(4)	0.4(5)	0.3(5)
C11	-0.1206(13)	0.4164(7)	0.2459(8)	10.7(7)	5.1(6)	4.9(5)	2.4(5)	3.7(5)	-0.1(4)
C12	-0.1686(11)	0.4125(7)	0.0670(8)	4.7(5)	5.9(6)	4.6(5)	1.2(4)	-0.4(4)	1.3(4)
C13	0.1261(9)	0.1046(6)	0.2641(6)	4.0(4)	3.9(4)	3.2(4)	1.1(4)	0.4(3)	0.3(4)
C14	0.1021(10)	0.0589(7)	0.3437(7)	3.9(5)	6.0(6)	4.3(5)	0.4(4)	0.2(4)	1.2(4)
C15	-0.0289(12)	0.0605(7)	0.3711(7)	6.5(6)	6.4(6)	3.4(4)	0.4(5)	1.0(4)	1.6(4)
C16	-0.1333(11)	0.1066(7)	0.3181(7)	5.6(5)	4.6(5)	4.9(5)	-0.4(4)	2.5(4)	1.0(4)
C17	-0.1603(10)	0.1497(7)	0.2390(7)	3.6(4)	4.5(5)	5.6(5)	-0.3(4)	0.9(4)	1.8(4)

The form of the anisotropic thermal parameter is:

$$\exp[-1/4(B_{11}h^2a^*2 + B_{22}k^2b^*2 + B_{33}l^2c^*2 + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}kla^*c^*)]$$

Table II. Bond Distances (Å) and Angles (Deg) in $W(OBu^t)_3(NO)(C_5H_5N)$ ^a

ATOMS			DISTANCE	ATOMS			DISTANCE
W	O1		1.876(6)	C1	C3		1.527(15)
W	O2		1.898(6)	C1	C4		1.547(15)
W	O3		1.887(6)	C5	C6		1.52(2)
W	N1		1.732(8)	C5	C7		1.533(15)
W	N2		2.323(7)	C5	C8		1.560(15)
O1	C1		1.476(11)	C9	C10		1.520(14)
O2	C5		1.449(11)	C9	C11		1.544(14)
O3	C9		1.446(11)	C9	C12		1.538(14)
O4	N1		1.250(10)	C13	C14		1.394(13)
N2	C13		1.318(11)	C14	C15		1.377(14)
N2	C17		1.343(11)	C15	C16		1.389(14)
C1	C2		1.53(2)	C16	C17		1.393(13)

ATOMS			ANGLE	ATOMS			ANGLE
O1	W	O2	117.3(3)	C2	C1	C3	111.(1)
O1	W	O3	115.7(3)	C2	C1	C4	113.(1)
O1	W	N1	100.7(3)	C3	C1	C4	112.(1)
O1	W	N2	80.4(3)	O2	C5	C6	108.3(9)
O2	W	O3	117.6(3)	O2	C5	C7	109.5(8)
O2	W	N1	100.9(3)	O2	C5	C8	104.3(8)
O2	W	N2	81.0(2)	C6	C5	C7	110.(1)
O3	W	N1	99.4(3)	C6	C5	C8	113.(1)
O3	W	N2	77.7(3)	C7	C5	C8	111.(1)
N1	W	N2	177.0(3)	O3	C9	C10	108.8(8)
W	O1	C1	135.9(6)	O3	C9	C11	104.4(8)
W	O2	C5	134.3(6)	O3	C9	C12	110.1(8)
W	O3	C9	136.0(5)	C10	C9	C11	110.(1)
W	N1	O4	179.2(8)	C10	C9	C12	111.6(9)
W	N2	C13	125.3(6)	C11	C9	C12	111.8(9)
W	N2	C17	116.0(6)	N2	C13	C14	122.7(9)
C13	N2	C17	118.7(8)	C13	C14	C15	118.8(9)
O1	C1	C2	107.4(8)	C14	C15	C16	119.1(9)
O1	C1	C3	108.9(8)	C15	C16	C17	118.(1)
O1	C1	C4	103.3(8)	N2	C17	C16	122.6(9)

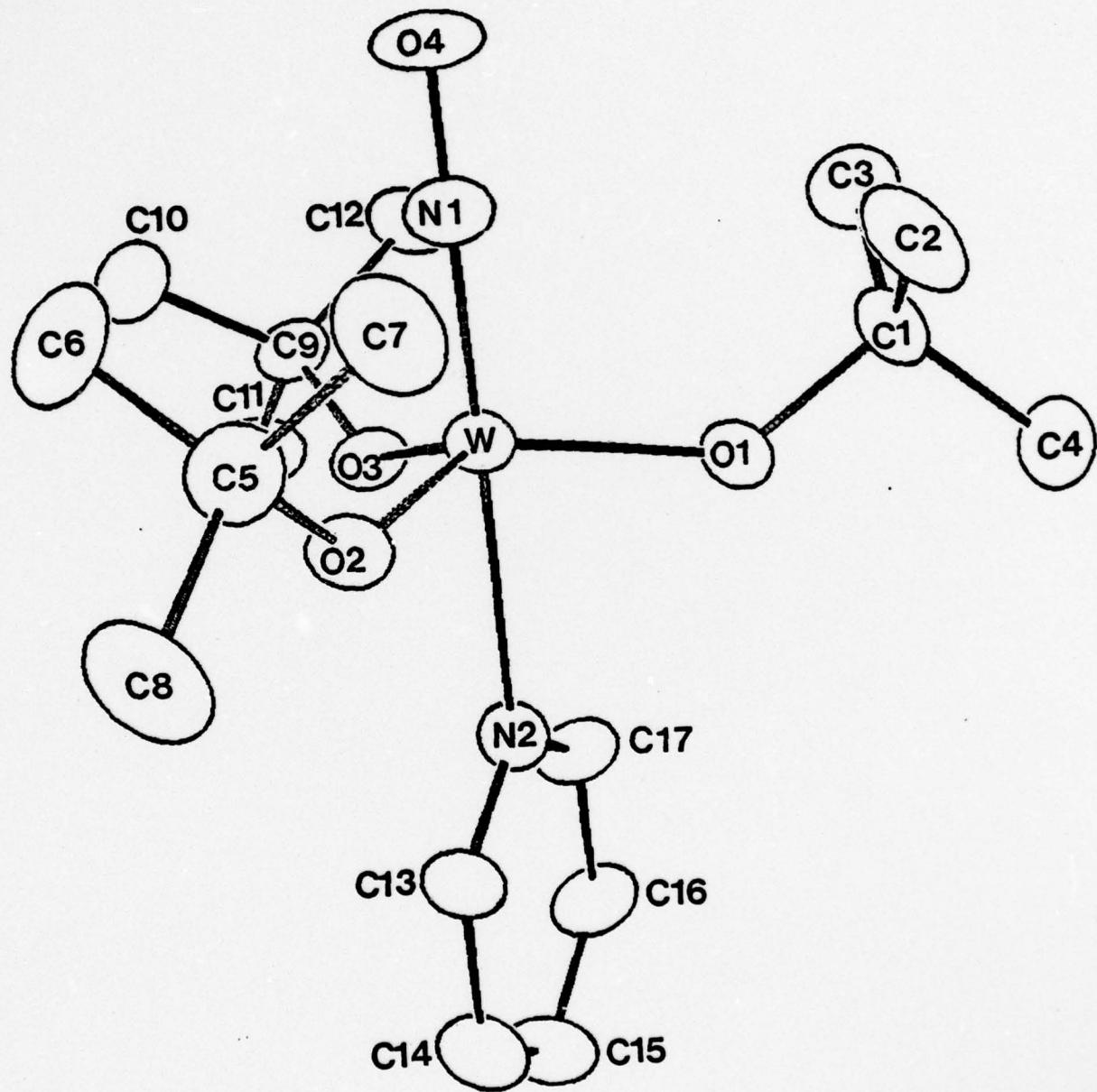
^aNumbers in parentheses are estimated standard deviations in the least significant digits.

Table III

Compound	M-NÅ	N-OÅ	M-N-O angle ⁰	ν(NO)cm ⁻¹	ref.
W(OBu) ^t ₃ (NO)(pyridine)	1.732(8)	1.25(1)	179.2(8)	1555	a
[Mo(OPr) ¹ ₃ NO] ₂	1.754(7)	1.19(1)	178(1)	1640	b
Cr(NSi ₂ Me ₆) ₃ NO	1.738(20)	1.191(28)	180 ¹	1698 ^j	c
Ru(η ³ -allyl)(NO)(PPh ₃) ₂	1.751(6)	1.188(8)	173.8(6)	1640	d
RuCl ₃ (NO)(PPh ₃) ₂	1.737(7)	1.142(8)	180 ¹	1876	e
RuCl ₃ (NO)(PPh ₂ Me) ₂	1.744(6)	1.132(6)	176.4(6)	1860 ^k	f
[RuBr ₃ (NO)(Et ₂ SO)] ₂	1.71(1)	1.16(1)	178(1)	1874	g
[Mo(CN) ₅ NO] ⁴⁻	1.95(3)	1.23(4)	175(3)	1455 ^l	h

^athis work; ^bref 4; ^cD. C. Bradley, M. B. Hursthouse, C. W. Newing and A. J. Welch, J.C.S. Chem. Commun., 567 (1972); ^dM. W. Schoonover and R. Eisenberg, J. Am. Chem. Soc. 99, 8371 (1977); ^eB. L. Haymore and J. A. Ibers, Inorg. Chem. 14, 3060 (1975); ^fA. J. Schultz, R. L. Henry and R. Eisenberg, Inorg. Chem. 13, 732 (1974); ^gJ. E. Fergusson, C. T. Page and W. T. Robinson, Inorg. Chem., 15, 2270 (1976); ^hD. H. Svedung and N.-G. Vannerberg, Acta. Chem. Scand. 22, 1551 (1968); ⁱcrystallographically imposed linearity; ^jC. W. Newing, Ph.D. Thesis London University 1971; ^kJ. Chatt and B. L. Shaw, J. Chem. Soc., A, 1811 (1966). ^lR. F. Riley and L. Ho, J. Inorg. Nucl. Chem. 24, 1121 (1962).

Figure 1. An ORTEP view of the $W(OBu^t)_3(NO)(C_5H_5N)$ molecule using 40% probability ellipsoids and showing the atom numbering scheme.



10*FOBS & 10*FCALC FOR W(0-T-BU)3(C5H5N) (NO)			COTTON, CHISHOLM ET AL 1978J			W(0-T-BU)3(C5H5N) (NO)			COTTON, CHISHOLM ET AL 1978J			
H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L
0	0	0	1548	1556	0	7	8	9	10	11	12	13
1800	1844	536	1792	1795	10	11	12	13	14	15	16	17
2184	644	1305	2295	2897	14	15	16	17	18	19	20	21
2452	7771	1583	1765	1727	14	15	16	17	18	19	20	21
2602	16159	1233	1714	1792	14	15	16	17	18	19	20	21
2771	1766	1212	1724	1794	14	15	16	17	18	19	20	21
2852	1776	1212	1732	1794	14	15	16	17	18	19	20	21
2952	1776	1212	1732	1794	14	15	16	17	18	19	20	21
3002	1776	1212	1732	1794	14	15	16	17	18	19	20	21
3144	1823	1212	1732	1794	14	15	16	17	18	19	20	21
3294	1832	1212	1732	1794	14	15	16	17	18	19	20	21
3434	1842	1212	1732	1794	14	15	16	17	18	19	20	21
3574	1852	1212	1732	1794	14	15	16	17	18	19	20	21
3714	1862	1212	1732	1794	14	15	16	17	18	19	20	21
3854	1872	1212	1732	1794	14	15	16	17	18	19	20	21
3994	1882	1212	1732	1794	14	15	16	17	18	19	20	21
4134	1892	1212	1732	1794	14	15	16	17	18	19	20	21
4274	1902	1212	1732	1794	14	15	16	17	18	19	20	21
4414	1912	1212	1732	1794	14	15	16	17	18	19	20	21
4554	1922	1212	1732	1794	14	15	16	17	18	19	20	21
4694	1932	1212	1732	1794	14	15	16	17	18	19	20	21
4834	1942	1212	1732	1794	14	15	16	17	18	19	20	21
4974	1952	1212	1732	1794	14	15	16	17	18	19	20	21
5114	1962	1212	1732	1794	14	15	16	17	18	19	20	21
5254	1972	1212	1732	1794	14	15	16	17	18	19	20	21
5394	1982	1212	1732	1794	14	15	16	17	18	19	20	21
5534	1992	1212	1732	1794	14	15	16	17	18	19	20	21
5674	2002	1212	1732	1794	14	15	16	17	18	19	20	21
5814	2012	1212	1732	1794	14	15	16	17	18	19	20	21
5954	2022	1212	1732	1794	14	15	16	17	18	19	20	21
6094	2032	1212	1732	1794	14	15	16	17	18	19	20	21
6234	2042	1212	1732	1794	14	15	16	17	18	19	20	21
6374	2052	1212	1732	1794	14	15	16	17	18	19	20	21
6514	2062	1212	1732	1794	14	15	16	17	18	19	20	21
6654	2072	1212	1732	1794	14	15	16	17	18	19	20	21
6794	2082	1212	1732	1794	14	15	16	17	18	19	20	21
6934	2092	1212	1732	1794	14	15	16	17	18	19	20	21
7074	2102	1212	1732	1794	14	15	16	17	18	19	20	21
7214	2112	1212	1732	1794	14	15	16	17	18	19	20	21
7354	2122	1212	1732	1794	14	15	16	17	18	19	20	21
7494	2132	1212	1732	1794	14	15	16	17	18	19	20	21
7634	2142	1212	1732	1794	14	15	16	17	18	19	20	21
7774	2152	1212	1732	1794	14	15	16	17	18	19	20	21
7914	2162	1212	1732	1794	14	15	16	17	18	19	20	21
8054	2172	1212	1732	1794	14	15	16	17	18	19	20	21
8194	2182	1212	1732	1794	14	15	16	17	18	19	20	21
8334	2192	1212	1732	1794	14	15	16	17	18	19	20	21
8474	2202	1212	1732	1794	14	15	16	17	18	19	20	21
8614	2212	1212	1732	1794	14	15	16	17	18	19	20	21
8754	2222	1212	1732	1794	14	15	16	17	18	19	20	21
8894	2232	1212	1732	1794	14	15	16	17	18	19	20	21
9034	2242	1212	1732	1794	14	15	16	17	18	19	20	21
9174	2252	1212	1732	1794	14	15	16	17	18	19	20	21
9314	2262	1212	1732	1794	14	15	16	17	18	19	20	21
9454	2272	1212	1732	1794	14	15	16	17	18	19	20	21
9594	2282	1212	1732	1794	14	15	16	17	18	19	20	21
9734	2292	1212	1732	1794	14	15	16	17	18	19	20	21
9874	2302	1212	1732	1794	14	15	16	17	18	19	20	21
10014	2312	1212	1732	1794	14	15	16	17	18	19	20	21
10154	2322	1212	1732	1794	14	15	16	17	18	19	20	21
10294	2332	1212	1732	1794	14	15	16	17	18	19	20	21
10434	2342	1212	1732	1794	14	15	16	17	18	19	20	21
10574	2352	1212	1732	1794	14	15	16	17	18	19	20	21
10714	2362	1212	1732	1794	14	15	16	17	18	19	20	21
10854	2372	1212	1732	1794	14	15	16	17	18	19	20	21
10994	2382	1212	1732	1794	14	15	16	17	18	19	20	21
11134	2392	1212	1732	1794	14	15	16	17	18	19	20	21
11274	2402	1212	1732	1794	14	15	16	17	18	19	20	21
11414	2412	1212	1732	1794	14	15	16	17	18	19	20	21
11554	2422	1212	1732	1794	14	15	16	17	18	19	20	21
11694	2432	1212	1732	1794	14	15	16	17	18	19	20	21
11834	2442	1212	1732	1794	14	15	16	17	18	19	20	21
11974	2452	1212	1732	1794	14	15	16	17	18	19	20	21
12114	2462	1212	1732	1794	14	15	16	17	18	19	20	21
12254	2472	1212	1732	1794	14	15	16	17	18	19	20	21
12394	2482	1212	1732	1794	14	15	16	17	18	19	20	21
12534	2492	1212	1732	1794	14	15	16	17	18	19	20	21
12674	2502	1212	1732	1794	14	15	16	17	18	19	20	21
12814	2512	1212	1732	1794	14	15	16	17	18	19	20	21
12954	2522	1212	1732	1794	14	15	16	17	18	19	20	21
13094	2532	1212	1732	1794	14	15	16	17	18	19	20	21
13234	2542	1212	1732	1794	14	15	16	17	18	19	20	21
13374	2552	1212	1732	1794	14	15	16	17	18	19	20	21
13514	2562	1212	1732	1794	14	15	16	17	18	19	20	21
13654	2572	1212	1732	1794	14	15	16	17	18	19	20	21
13794	2582	1212	1732	1794	14	15	16	17	18	19	20	21
13934	2592	1212	1732	1794	14	15	16	17	18	19	20	21
14074	2602	1212	1732	1794	14	15	16	17	18	19	20	21
14214	2612	1212	1732	1794	14	15	16	17	18	19	20	21
14354	2622	1212	1732	1794	14	15	16	17	18	19	20	21
14494	2632	1212	1732	1794	14	15	16	17	18	19	20	21
14634	2642	1212	1732	1794	14	15	16	17	18	19	20	21
14774	2652	1212	1732	1794	14	15	16	17	18	19	20	21
14914	2662	1212	1732	1794	14	15	16	17	18	19	20	21
15054	2672	1212	1732	1794	14	15	16	17	18	19	20	21
15194	2682	1212	1732	1794	14	15	16	17	18	19	20	21
15334	2692	1212	1732	1794	14	15	16	17	18	19	20	21
15474	2702	1212	1732	1794	14	15	16	17	18	19	20	21
15614	2712	1212	1732	1794	14	15	16	17	18	19	20	21
15754	2722	1212	1732	1794	14	15	16	17	18	19	20	21
15894	2732	1212	1732	1794	14	15	16	17	18	19	20	21
16034	2742	1212	1732	1794	14	15	16	17	18	19	20	21
16174	2752	1212	1732	1794	14	15	16	17	18	19	20	21
16314	2762	1212	1732	1794	14	15	16	17	18	19	20	21
16454	2772	1212	1732	1794	14	15	16	17	18	19	20	21
16594	2782	1212	1732	1794	14	15	16	17	18	19	20	21
16734	2792	1212	1732	1794	14	15	16	17	18	19	20	21
16874	2802	1212	1732	1794	14	15	16	17	18	19	20	21
16914	2812	1212	1732	1794	14	15	16	17	18	19	20	21
17054	2822	1212	1732	1794	14	15	16	17	18	19	20	21
17194	2832	1212	1732	1794	14	15	16	17	18			

100*FO86 & 100*FCALC FOR μ (0-T-BU)₃(CSH5N) (NO) (COTTON, CHISHOLM ET AL 1978)

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10*FOBS 10*CALE FOR W68-I-BW) 3 (C5HSN) AND EDITION: CHISHOLM ET AL 1927

BEST AVAILABLE COPY

10*FOBS & 10*FCALC FOR W(0-T-BU)3(C5H5N) (NO) [COTTON; CHISHOLM ET AL 1978]									
H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
5	7	8	894	894	9	9	19	342	342
6	7	10	8918	884	6	6	6	668	668
6	7	11	684	674	6	6	6	658	654
6	7	12	628	623	6	6	6	654	649
6	7	13	343	353	6	6	6	649	649
6	7	14	573	562	6	6	6	649	649
6	7	15	373	362	6	6	6	649	649
6	7	16	478	475	6	6	6	649	649
6	7	17	239	250	6	6	6	649	649
6	7	18	1109	1106	6	6	6	649	649
6	7	19	1527	1492	6	6	6	649	649
6	7	20	596	599	6	6	6	649	649
6	7	21	1158	1165	6	6	6	649	649
6	7	22	749	685	6	6	6	649	649
6	7	23	939	938	6	6	6	649	649
6	7	24	485	485	6	6	6	649	649
6	7	25	965	965	6	6	6	649	649
6	7	26	921	921	6	6	6	649	649
6	7	27	689	689	6	6	6	649	649
6	7	28	758	758	6	6	6	649	649
6	7	29	231	231	6	6	6	649	649
6	7	30	917	917	6	6	6	649	649
6	7	31	2139	2139	6	6	6	649	649
6	7	32	359	359	6	6	6	649	649
6	7	33	648	648	6	6	6	649	649
6	7	34	1161	1161	6	6	6	649	649
6	7	35	699	699	6	6	6	649	649
6	7	36	682	682	6	6	6	649	649
6	7	37	479	479	6	6	6	649	649
6	7	38	414	414	6	6	6	649	649
6	7	39	818	818	6	6	6	649	649
6	7	40	452	452	6	6	6	649	649
6	7	41	439	439	6	6	6	649	649
6	7	42	759	759	6	6	6	649	649
6	7	43	344	344	6	6	6	649	649
6	7	44	751	751	6	6	6	649	649
6	7	45	466	466	6	6	6	649	649
6	7	46	444	444	6	6	6	649	649
6	7	47	444	444	6	6	6	649	649
6	7	48	408	408	6	6	6	649	649
6	7	49	271	271	6	6	6	649	649
6	7	50	531	531	6	6	6	649	649
6	7	51	556	556	6	6	6	649	649
6	7	52	628	628	6	6	6	649	649
6	7	53	494	494	6	6	6	649	649
6	7	54	609	609	6	6	6	649	649
6	7	55	379	379	6	6	6	649	649
6	7	56	344	344	6	6	6	649	649
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6	7	58	379	379	6	6	6	649	649
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6	7	64	379	379	6	6	6	649	649
6	7	65	379	379	6	6	6	649	649
6	7	66	379	379	6	6	6	649	649
6	7	67	379	379	6	6	6	649	649
6	7	68	379	379	6	6	6	649	649
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6	7	70	379	379	6	6	6	649	649
6	7	71	379	379	6	6	6	649	649
6	7	72	379	379	6	6	6	649	649
6	7	73	379	379	6	6	6	649	649
6	7	74	379	379	6	6	6	649	649
6	7	75	379	379	6	6	6	649	649
6	7	76	379	379	6	6	6	649	649
6	7	77	379	379	6	6	6	649	649
6	7	78	379	379	6	6	6	649	649
6	7	79	379	379	6	6	6	649	649
6	7	80	379	379	6	6	6	649	649
6	7	81	379	379	6	6	6	649	649
6	7	82	379	379	6	6	6	649	649
6	7	83	379	379	6	6	6	649	649
6	7	84	379	379	6	6	6	649	649
6	7	85	379	379	6	6	6	649	649
6	7	86	379	379	6	6	6	649	649
6	7	87	379	379	6	6	6	649	649
6	7	88	379	379	6	6	6	649	649
6	7	89	379	379	6	6	6	649	649
6	7	90	379	379	6	6	6	649	649
6	7	91	379	379	6	6	6	649	649
6	7	92	379	379	6	6	6	649	649
6	7	93	379	379	6	6	6	649	649
6	7	94	379	379	6	6	6	649	649
6	7	95	379	379	6	6	6	649	649
6	7	96	379	379	6	6	6	649	649
6	7	97	379	379	6	6	6	649	649
6	7	98	379	379	6	6	6	649	649
6	7	99	379	379	6	6	6	649	649
6	7	100	379	379	6	6	6	649	649
6	7	101	379	379	6	6	6	649	649
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6	7	103	379	379	6	6	6	649	649
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6	7	105	379	379	6	6	6	649	649
6	7	106	379	379	6	6	6	649	649
6	7	107	379	379	6	6	6	649	649
6	7	108	379	379	6	6	6	649	649
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10*FOBS & 10*FCALC FOR W(0-T-80)3(C5H5N) (IND) COTTON, CHISHOLM ET AL 19781												PAGE			
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10*FOBS & 10*FCALC FOR W(O-T-BU)3(C5H5N) (NO)		COTTON, CHISHOLM ET AL 1978]		PAGE 9	
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